AL-TR-90-028

AD:

(2)

Final Report
for the period
9 February 1988
to 26 June 1990

# Construction of Flowing Afterglow Apparatus



December 1990

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F04611-88-C-0023

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*Prepared for the:* **Astronautics Laboratory (AFSC)**
Air Force Space Technology Center
Space Systems Division
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## FOREWORD

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# REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

| | |
|---|---|
| 1a. REPORT SECURITY CLASSIFICATION: UNCLASSIFIED | 1b. RESTRICTIVE MARKINGS |
| 2a. SECURITY CLASSIFICATION AUTHORITY | 3. DISTRIBUTION/AVAILABILITY OF REPORT: Approved for Public Release; Distribution is Unlimited |
| 2b. DECLASSIFICATION/DOWNGRADING SCHEDULE | |
| 4. PERFORMING ORGANIZATION REPORT NUMBER(S) | 5. MONITORING ORGANIZATION REPORT NUMBER(S): AL-TR-90-028 |
| 6a. NAME OF PERFORMING ORGANIZATION: Extrel Corporation; 6b. OFFICE SYMBOL (If applicable) | 7a. NAME OF MONITORING ORGANIZATION: Astronautics Laboratory (AFSC) |
| 6c. ADDRESS (City, State, and ZIP Code): 575 Epsilon Drive, Pittsburgh PA 15238 | 7b. ADDRESS (City, State, and ZIP Code): AL/LSXP, Edwards AFB CA 93523-5000 |
| 8a. NAME OF FUNDING/SPONSORING ORGANIZATION; 8b. OFFICE SYMBOL (If applicable) | 9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: F04611-88-C-0023 |
| 8c. ADDRESS (City, State, and ZIP Code) | 10. SOURCE OF FUNDING NUMBERS |

| PROGRAM ELEMENT NO. | PROJECT NO. | TASK NO | WORK UNIT ACCESSION NO. |
|---|---|---|---|
| 65502F | 5730 | 006T | 344406 |

11. TITLE (Include Security Classification): Construction of Flowing Afterglow Apparatus (U)

12. PERSONAL AUTHOR(S): Dheandhanoo, Seksan and Fite, Wade

| 13a. TYPE OF REPORT | 13b. TIME COVERED | 14. DATE OF REPORT (Year, Month, Day) | 15. PAGE COUNT |
|---|---|---|---|
| Final | FROM 880209 TO 900625 | 9012 | 19 |

16. SUPPLEMENTARY NOTATION

| 17. COSATI CODES | | | 18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) |
|---|---|---|---|
| FIELD | GROUP | SUB-GROUP | Flowing afterglow, flow tubes, gas-phase chemical kinetics |
| 20 | 11 | | |
| | | | |

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The design and construction of a Flowing Afterglow (FA) apparatus were undertaken in the Phase II SBIR project, F04611-88-C-0023. The FA apparatus consists of three basic sections: Flow tubes, a detection system and a Vacuum/Gas Handling system. This instrument can be used for gas-phase chemical kinetics studies of both ion-neutral and neutral-neutral reactions. The instrument was designed in a modular manner so that it can be easily assembled for appropriate experiments. A mechanical chopper is added to the system so that modulated molecular beam experiments can be performed. The detection system, which is a Triple Quadrupole Mass Spectrometer, can be used without the flow tubes as an analytical tool.

| 20. DISTRIBUTION/AVAILABILITY OF ABSTRACT | 21. ABSTRACT SECURITY CLASSIFICATION |
|---|---|
| ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS | UNCLASSIFIED |

| 22a. NAME OF RESPONSIBLE INDIVIDUAL | 22b. TELEPHONE (Include Area Code) | 22c. OFFICE SYMBOL |
|---|---|---|
| PETER J. DOLAN, CAPTAIN, USAF | (805) 275-5760 | LSXP |

DD Form 1473, JUN 86 — Previous editions are obsolete. — SECURITY CLASSIFICATION OF THIS PAGE

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## INTRODUCTION

The phase II project under contract number No. F04611-88-C-0023 was awarded to EXTREL in May 1988. The contract work involved construction of a Flowing Afterglow (FA) instrument followed the design work which was done under the Phase I project, Contract No. F04611-87-C-0043.

Shortly after Phase II work was awarded, EXTREL personnel (Dr. Seksan Dheandhanoo and Dr. Wade L. Fite) visited Capt. W. J. Lauderdale and Enfitek personnel at the Air Force Astronautics Laboratory (AFAL) at Edwards Air Force Base to discuss the construction and testing plan and to inspect the laboratory in which the apparatus will be located.

Most FA instruments were specifically designed for chemical kinetic studies of either ion-neutral or neutral-neutral reactions. The design of this FA instrument was directed toward a basic instrument on which either type of experiment could be performed. The complete instrument is very much like the Phase I design. Some minor modifications were requested by Edwards Air Force personnel during the course of construction. These modifications were necessary for the integration of the EXTREL instrument to the Enfitek instrument and to fit the location in the laboratory. At the kick-off meeting we learned that certain future experiments may involve the use of corrosive gas. Therefore, fomblin pump oil was used in the mechanical pumps instead of regular oil. The turbomolecular pump was also equipped with a special part for corrosive gases. Details of the modifications were described in progress reports.

The FA instrument was shipped to the Astronautics Laboratory at Edwards Air Force Base in October, 1989 after shake down testing at EXTREL. The installation of the instrument was completed at the end of October, 1989.

## FLOWING AFTERGLOW (FA) INSTRUMENT

The FA instrument is composed of 3 main sections: the flow tubes, the detection system, and the vacuum/gas handling system. The diagram of the overall instrument is shown in Fig. 1. To provide versatility, the FA instrument was designed in a modular manner so that it can be easily assembled for appropriate experiments. The FA instrument will be described in detail in the manual. This report contains only a brief description of the instrument.

### Flow Tubes

The flow tube is the section in which chemical reaction occurs. Due to the differences in nature of ion-neutral (I-N) and neutral-neutral (N-N) reactions, two types of flow tubes were provided.

The I-N flow tube was made of stainless steel tubing of 3.3" in diameter. The I-N flow tube consists of several sections. These sections may be readily assembled in different configurations to accommodate different types of experiments.

The N-N flow tubes were made of glass tubing. Three different sizes of glass flow tubes were built. The smallest glass flow tube is 28 mm in outer diameter (OD). This is the size of N-N flow tube that has been most commonly used by scientists. The other two glass flow tubes are 55 mm and 80 mm in OD. The experiments on the larger diameter flow tubes will provide information on the wall effect.

### Vacuum and Gas Handling Systems

#### Vacuum system

The gases from the flow tube are pumped out through a Leybold Heraeus Roots blower. The maximum pumping capacity of the blower is about 2500 CFM at 10 torr. Only a small portion of the gases is sampled through a small orifice. The sampled gases are ionized and analyzed by a detection system which is located inside one of the vacuum chambers.

The vacuum system of this instrument can be divided into 3 sections: the outer chamber, the middle chamber and the inner chamber. The outer chamber takes the gas flow from the flow tube and transmits it to the Roots blower. An 8" motor-driven gate valve was installed between the blower and the outer chamber. The function of this gate valve is to throttle the flow rate of the gas in the flow tubes.

A small portion of the gases from the flow tube flows into the middle chamber through a small orifice and forms a molecular beam. Pressure in the middle chamber is maintained at about $1 \times 10^{-4}$ torr by an Edwards diffusion pump with a pumping speed of ~ 800 l/s.

The detection system is located inside the inner chamber which is evacuated by a Leybold Heraeus turbomolecular pump with a pumping speed of 450 l/s. Typical pressure of this chamber is less than $1 \times 10^{-6}$ torr.

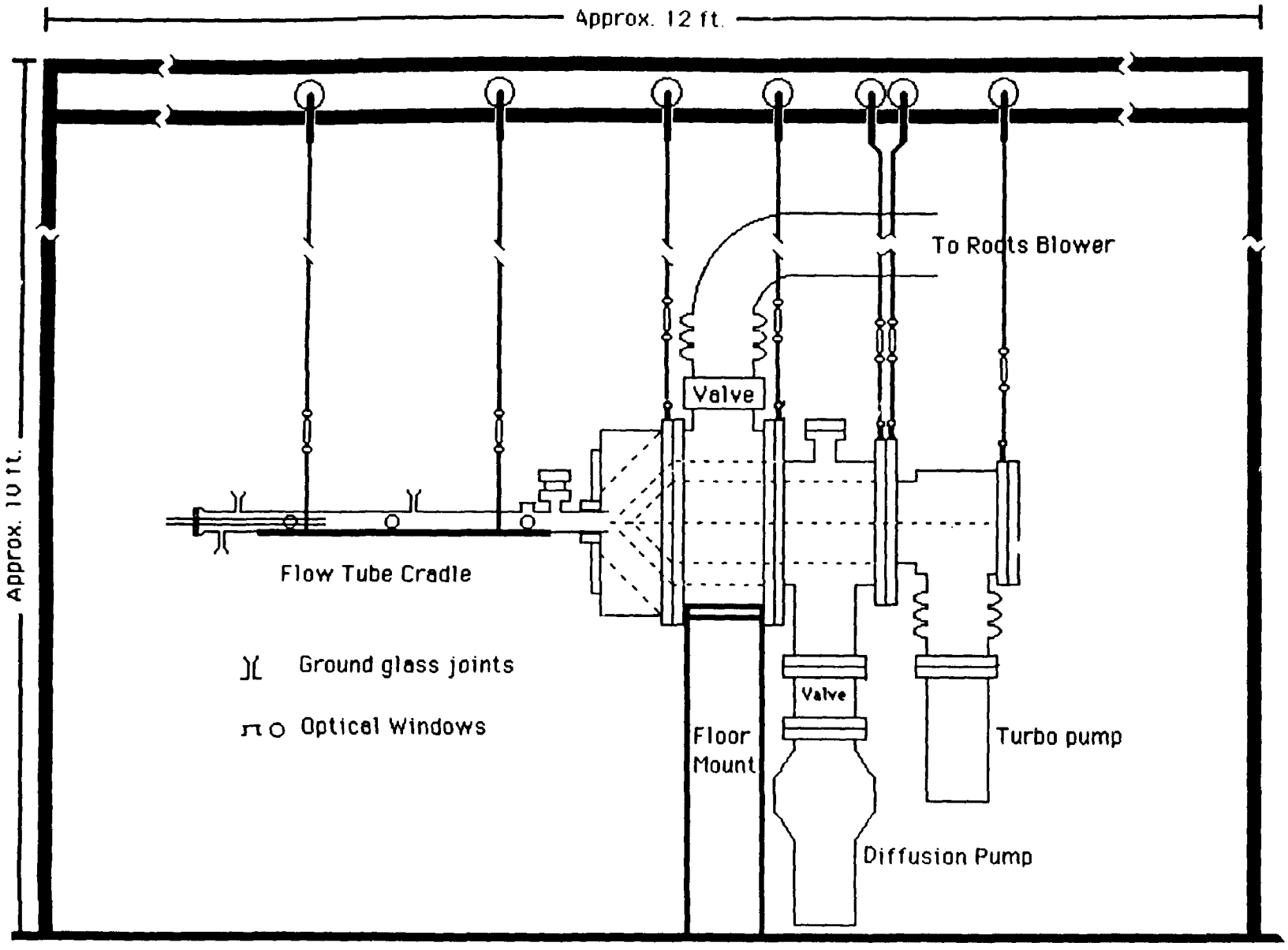


Fig. 1 Overview

Edwards mechanical pumps, model E2M12, were used as backing pumps for the diffusion pump and the turbomolecular pump. The pressure inside the middle and inner cone are monitored by ionization gauges.

This vacuum system was designed such that it is self-protecting. In case any of the following situations occur, the pumps will turn off automatically. These situations are: 1) power failure; 2) water shortage; 3) lack of air pressure for pneumatic valves; and 4) if pressure in the vacuum chamber is above the setting limit. Under normal circumstances, the pressure of the middle chamber is at least an order of magnitude higher than that of the inner chamber. Therefore, we usually set the vacuum system so that it will turn itself off when the pressure inside the middle chamber is higher than $5 \times 10^{-4}$ torr.

Once the system is shut down, it will not turn on automatically. The start up procedure is quite simple and it takes about 15 minutes to get the system back to the operating condition. The start up requires an operator to turn on a starting switch and all the pumps will be turned on sequentially by electronic interlocks. The system was designed to operate in this manner for safety purposes.

There are two operation modes for the vacuum system: the manual mode and the computer-controlled mode. In the manual mode, the system is turned on and off by an operator and it is a self-protecting system, as explained above. The electronics of the vacuum system were designed so that it can also be controlled by a computer. There is a switch in the back of the control panel for mode selection.

Gas handling system

The gas handling system involves the introduction of reactant gases and carrier gases into flow tubes. The gas handling system includes two types of gas manifolds. One is made of metal and will be used for non-corrosive gases. The other gas manifold is made of glass and will be used for corrosive gases. The metal gas manifold is connected to an Edwards mechanical pump. The function of the mechanical pump is to evacuate the gas manifold. All gases are introduced into flow tubes via MKS flow controllers. The flow rate of each gas can be easily adjusted or set by operators.

The selection of gas purification techniques depends on the nature of the gases. We installed two cold traps between the gas tanks and the flow meters. The cold trap is generally used to trap water and other impurities that have freezing points above the temperature of the cold trap.

Detection System

The function of the detection system is to analyze both ions and neutral species from the flow tubes. The detection system of this instrument includes a Triple Quadrupole Mass Spectrometer (TQMS), an electron impact ionizer, a mechanical molecular beam chopper and an electron multiplier.

Triple quadrupole mass spectrometer (TQMS)

The TQMS was selected as the main detection system. The TQMS consists of three single 3/4" quadrupole mass spectrometers, as shown in Fig. 2. The first and the third quadrupole can be operated as a single mass analyzer. Both quadrupoles are capable of analyzing ions of mass as high as 300 amu. The second quadrupole acts as a collision cell and can not be used as a mass analyzer. The TQMS can be either controlled manually or by a computer.

In most experiments, the TQMS will be used as a single quadrupole, which means either the first or the third quadrupole will be operated as a mass analyzer, the other two quadrupoles will be used as ion pipes.

The TQMS will be operated as a triple quadrupole in cases where there are more than one chemical species that have the same mass but differences in structure and composition. In such cases, the ions of interest, the parent ions, will be selected by the first quadrupole. The selected ions will then undergo collisions with a collision gas (normally Argon) in the second quadrupole. The fragment ions or so called daughter ions will be analyzed by the third quadrupole. In most cases the daughter spectra of different chemical species are not the same. By analyzing the daughter spectra, the parent ions can be identified. For instance, both CO and $N_2$ have mass of 28 amu. Daughter ions of CO are ions of mass 12 and 16 which correspond to $C^+$ and $O^+$ and the daughter ion of $N_2^+$ is ion of mass 14 which corresponds to $N^+$.

Electron impact ionizer

The electron impact ionizer was installed in front of the TQMS, see Fig. 2. Neutral chemical species from the flow tubes are ionized by means of electron impact ionization prior to being analyzed by the TQMS. For ion-molecule reaction experiments, ions from the flow tube are monitored instead of neutral species. In such cases, the electron gun in the ionizer is turned off and the ionizer itself behaves as a set of electrostatic lenses.

Mechanical chopper

A mechanical chopper with a frequency of 200 Hz is located in front of the inner cone. The chopper will be used in molecular beam experiments. Ions formed from the background gas can be separated from those formed from the neutral species from the flow tubes by using the chopper. Once the chopper is turned on, the molecular beam of chemical species from the flow tube will be periodically interrupted at the frequency of 200 Hz which results in the 200 Hz AC ion signal of specific phase. The ion signal from the backgrour d gas in the chamber is a DC signal. By using phase-sensitive AC signal detection with a lock-in amplifier, the chemical species from the flow tube can be easily separated from the background gas.

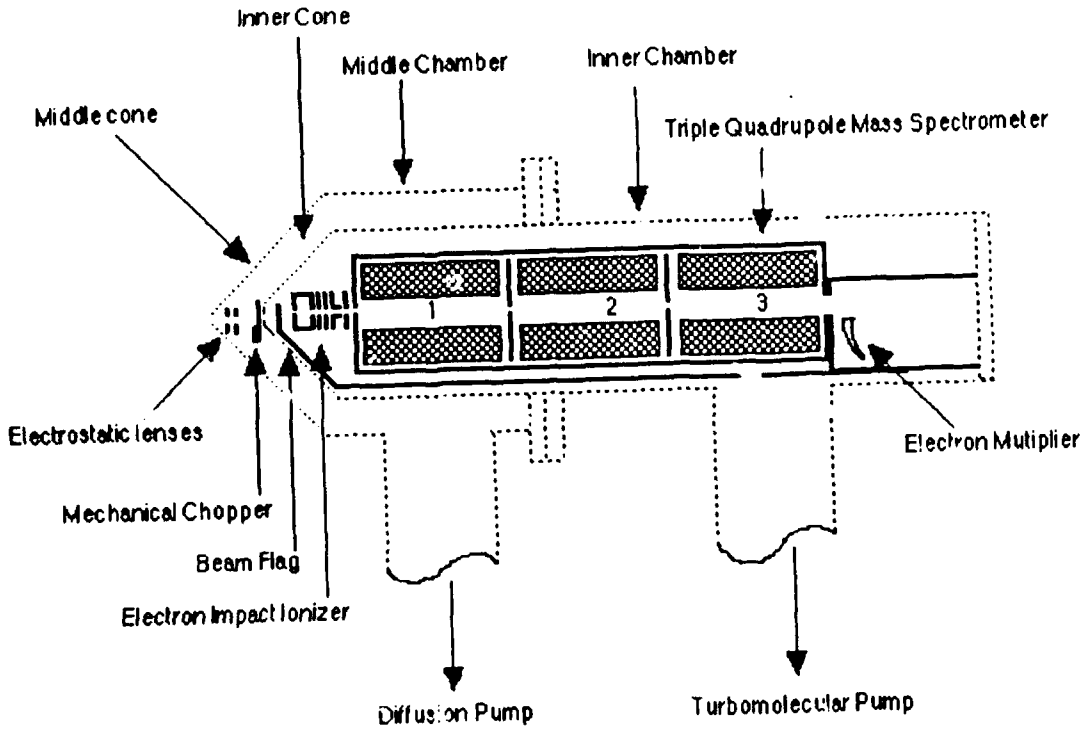


Fig. 2 Diagrams of parts inside the vacuum chambers

Electron multiplier

Ions from the TQMS are steered into an electron multiplier which is located behind the TQMS. This multiplier can be used to detect both positive and negative ions. The multiplier is equipped with a conversion dynode which increases the sensitivity of the multiplier for high mass ions. The multiplier can be operated in either the analog or counting mode. The counting mode is more sensitive than the analog mode and is normally used when the number density of ions is very low

Data acquisition

The FA system is equipped with a Televent data system, model1050-A Vector/One and a 386/20E Compaq personal computer (PC), model 40. The TQMS can be controlled manually or by the computer. This computer will be used to monitor ion signal and analyze mass spectra.

Control panels

All of the control and read-out panels were mounted on three racks, as shown in Fig. 3. All three racks are movable; therefore, they can be set at any location for the convenience of the operators.

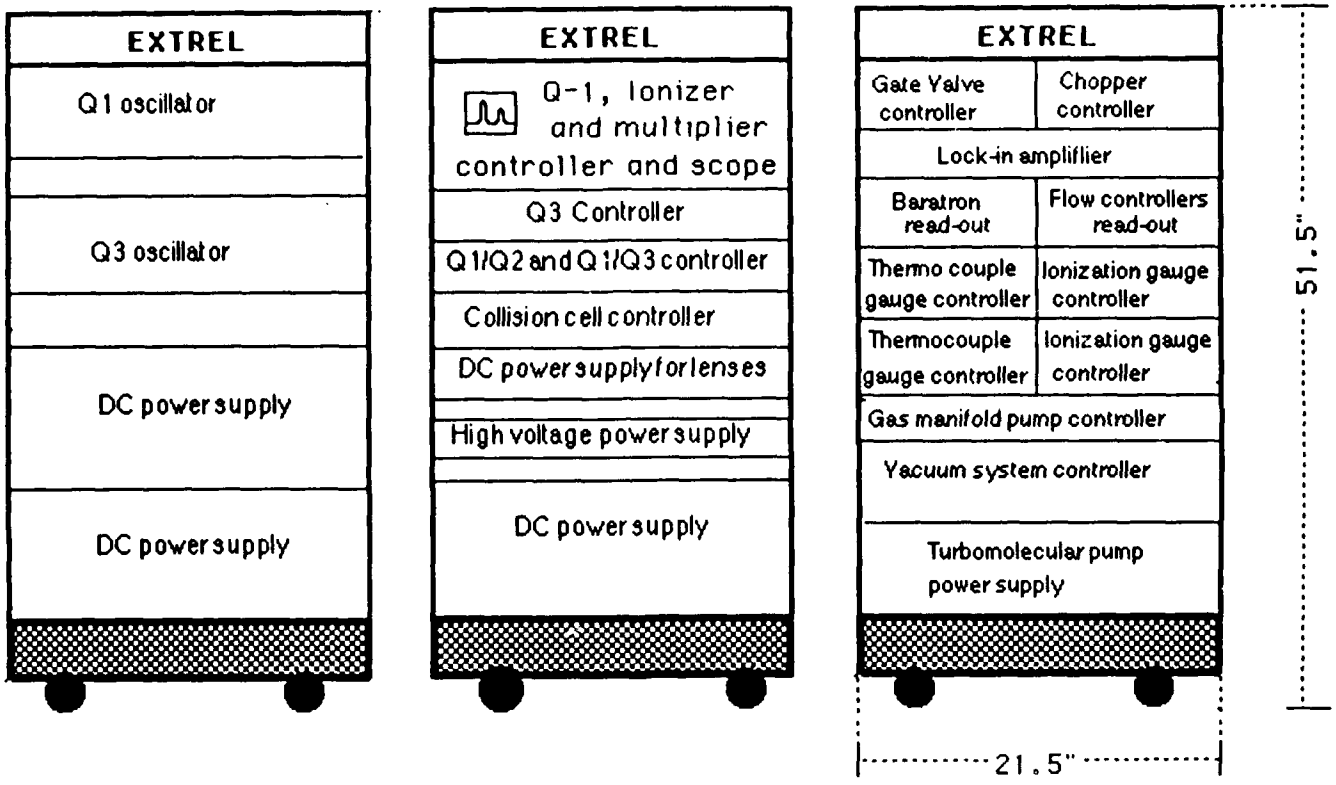


Fig. 3 Diagram of electronic racks

## FA INSTRUMENT TESTING

### Testing at EXTREL

The FA instrument was assembled at EXTREL for testing prior to the shipment to Edwards Air Force Base. We tested the over-all instrument by measuring the reaction rate coefficient of the reaction of He ions with nitrogen molecules. An example of the experimental data is shown in Fig. 4. The rate coefficient can be determined from the slope of the decay of helium ions. Details of the calculations are described in the opertor's manual. The rate coefficient obtained from this experiment is $1.6 \times 10^{-9}$ cc/sec. which agrees well with previous work done by other scientists.

A modulated beam experiment was performed during the testing. We were able to generate nitrogen atoms by using a microwave discharge.of the 3:1 mixture of nitrogen and helium. The microwave discharge was operated between 15 and 60 Watts.. The partially dissociated beam of nitrogen atoms and molecules from the 1" glass flow tube were ionized by electron impact ionization prior to being analysed by the TQMS. The energy of the electron was between 25 to 30 electron volts, which was sufficient to ionize both nitrogen atom and nitrogen molecules but not high enough to produce $N^+$ ions by dissociative ionization of the molecules. Therefore, the ions of mass 14 were most likely to be nitrogen atoms from the flow tube. The intensities of ions of mass 14 increased as the microwave power increased, see Fig. 5.

### Testing at Edwards Air Force Base

Dr. Seksan Dheandhanoo and a technician spent two weeks assembling the FA system at EAFB in October, 1989 Training at EAFB was undertaken in November, 1989. A major portion of this training session involved teaching EAFB personnel to operate the TQMS. We also used the TQMS to study the daughter spectrum of azide ions ($N_3H^+$) as part of the training. In this experiment, azide ions were produced by electron impact ionization. The ions of mass 43 amu, which correspond to azide ions , were selected by the first quadrupole and underwent collision with argon gas in the collision cell. The daughter ions were analysed by the third quadrupole. The daughter ions of azide are ions of masses 15 and 29 amu, which correspond to $NH^+$ and $N_2H^+$, respectively.

The reaction of $He^+$ ion with $O_2$ molecules was investigated using the I-N flow tube. The experimental data are shown in Fig. 6. The reaction rate coefficient obtained from the experiment is $1.19 \times 10^{-9}$ cc/sec which agree with previous results which were $0.9\text{-}1.5 \times 10^{-9}$ cc/sec

The integration of the EXTREL and Enfitek instruments was completed in December, 1989. We installed the Enfitek laser ablation source at the end of Extrel's I-N flow tube. A YAG laser which was supplied by Enfitek was used to generate aluminum atoms and ions. We were able to observe both aluminum atoms and ions.

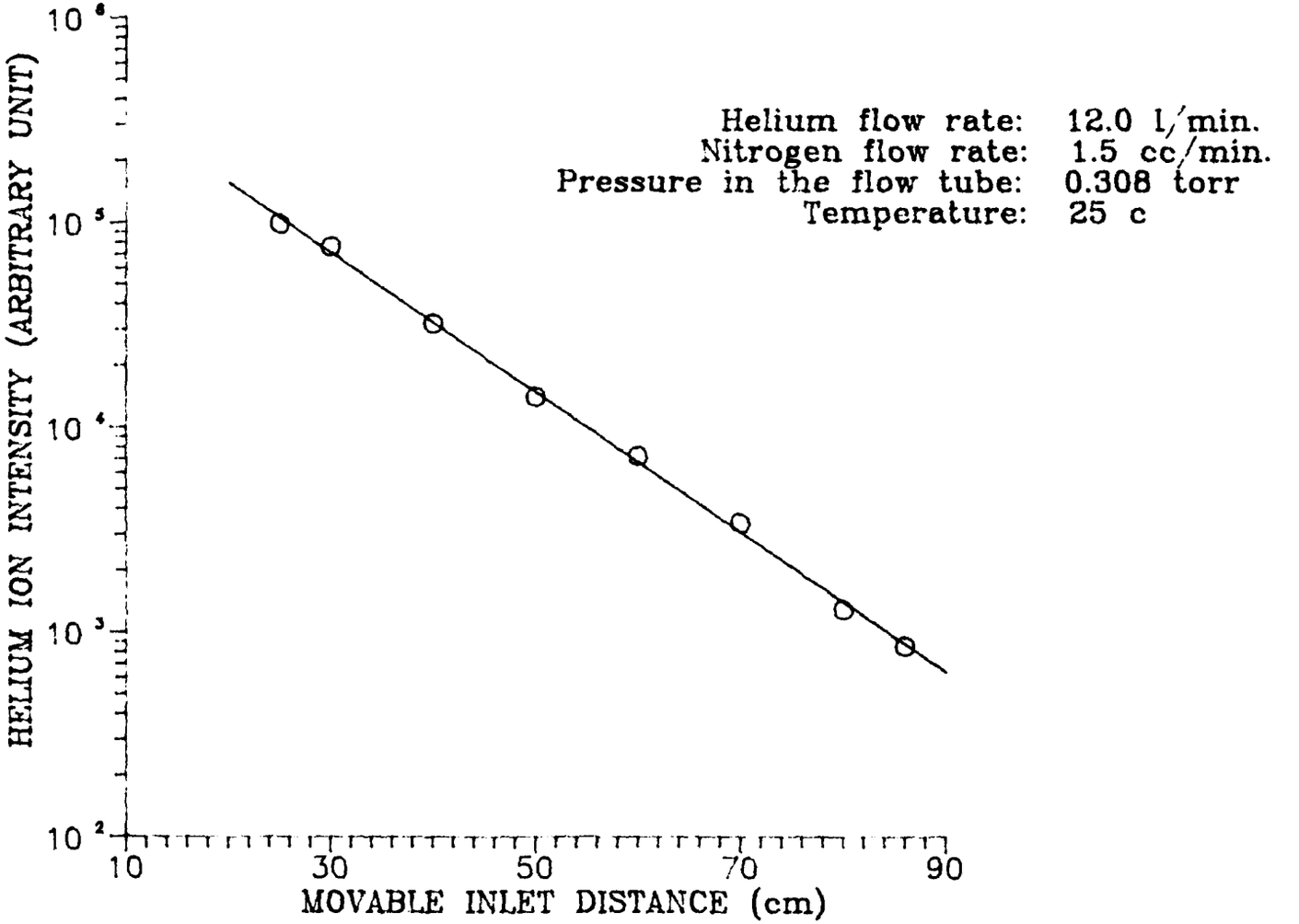


Fig. 4 Decay of He ions as a function of reaction distance.

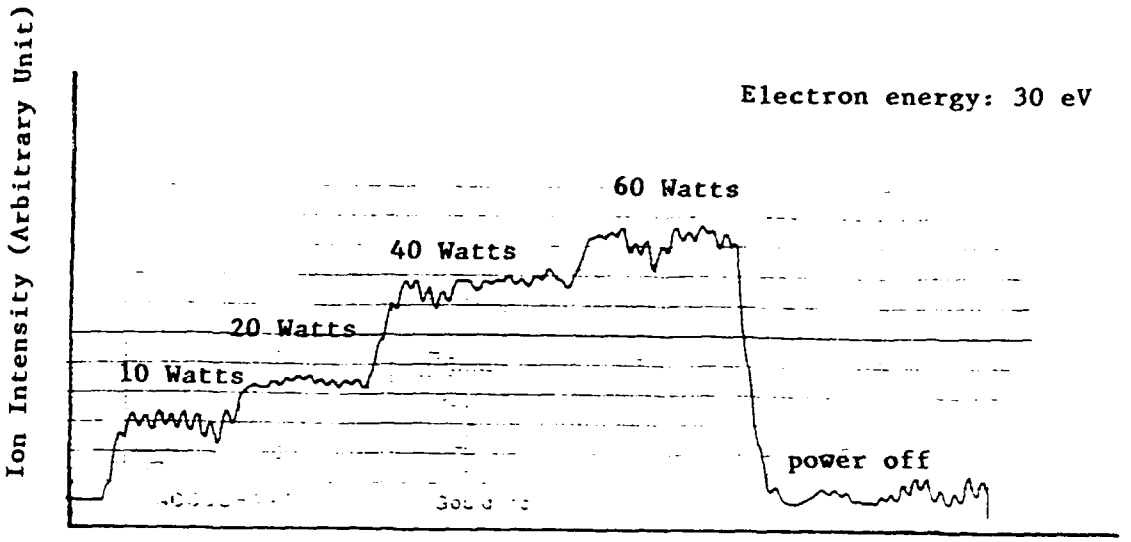


Fig.5. Ion intensities of $N^+$ ions as a function of microwave power.

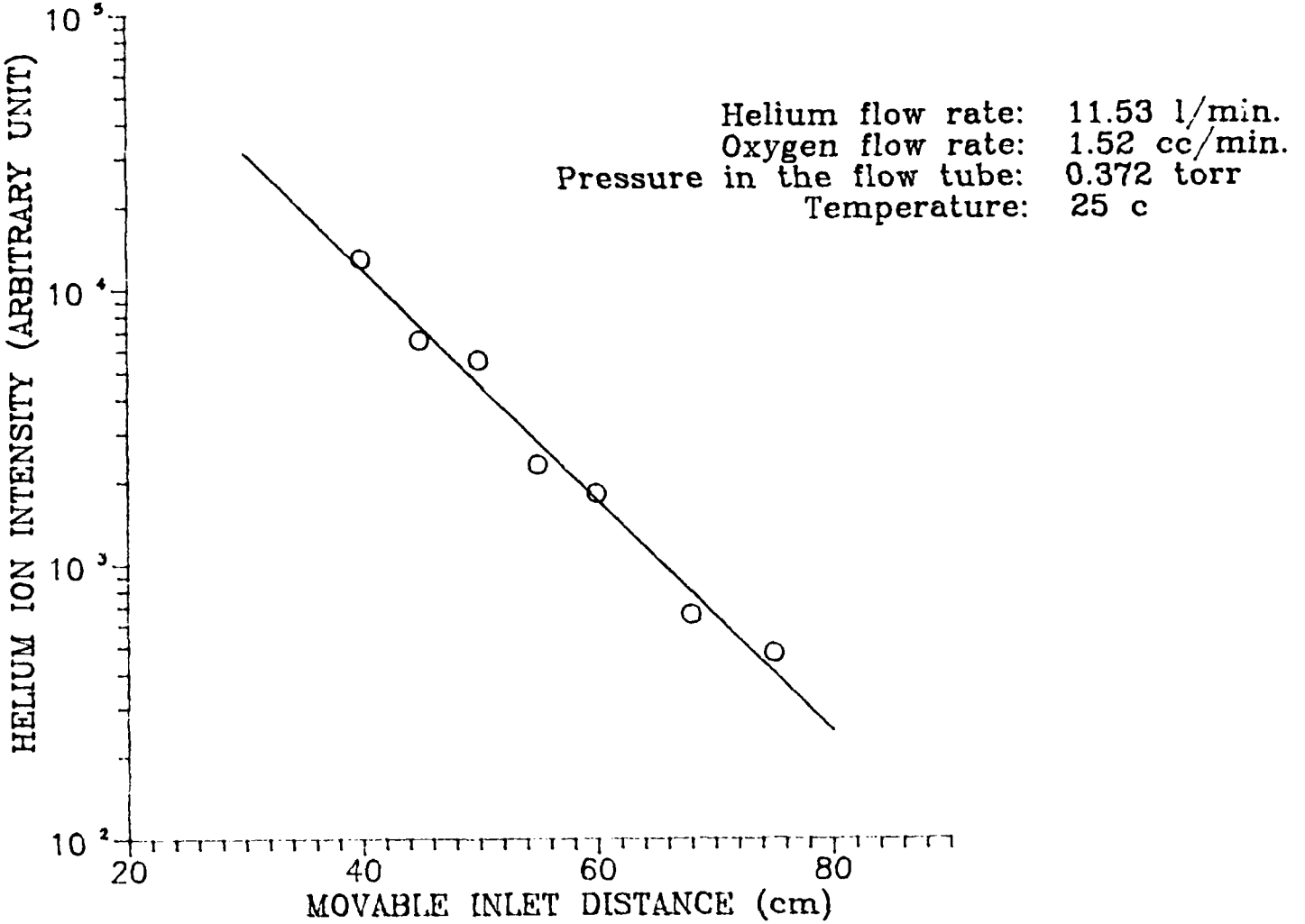


Fig. 6 Decay of He ions as a function of reaction distance.

The last training session was in early February, 1990. Dr. Dheandhanoo corrected a few final problems and determined that the FA system worked properly. Dr. Dheandhanoo and EAFB personnel attempted an experiment on the reaction of metastable $N_2^*$ ($^3\Sigma_u$) with azide. Argon was used as the carrier gas and $N_2^*$ was generated by the reaction of metastable Ar atoms with nitrogen molecules. The metastable Ar atoms were produced by a DC discharge of argon gas. A small amount of nitrogen gas (~20% of argon gas) was added to the flow tube at about 5 " downstream from the DC discharge. Attempts were made to detect $N_2^*$ molecules using laser spectroscopy and mass spectroscopy. However, neither techniques revealed the presence of $N_2^*$ in the flow tube.

According to the potential energy curves of $N_2$ and $N_2^+$ (see Fig. 7), it is possible to generate ground state $N_2^+$ from $N_2^*$ with electrons of energy slightly less than 14 eV. Since the ionization potential of ground state $N_2$ is 15.58 eV., we should be able to selectively ionized $N_2^*$ without ionizing ground state $N_2$. However, in this experiment, ions of mass 28 amu ($N_2^+$) were not observed unless the energy of electrons in the ionizer was higher than 17 eV. It was very likely that the $N_2^+$ ions came from ground state $N_2$ rather than $N_2^*$. This led us to believe that only a small amount of $N_2^*$ was generated and the loss of $N_2^*$ due to reactions with impurities in argon gas, such as water, was significant. We expected that $N_2^*$ also collided with the wall of the flow tube. To reduce the loss of $N_2^*$, future experiments should be conducted on a shorter flow tube. Further investigation of this reaction is needed.

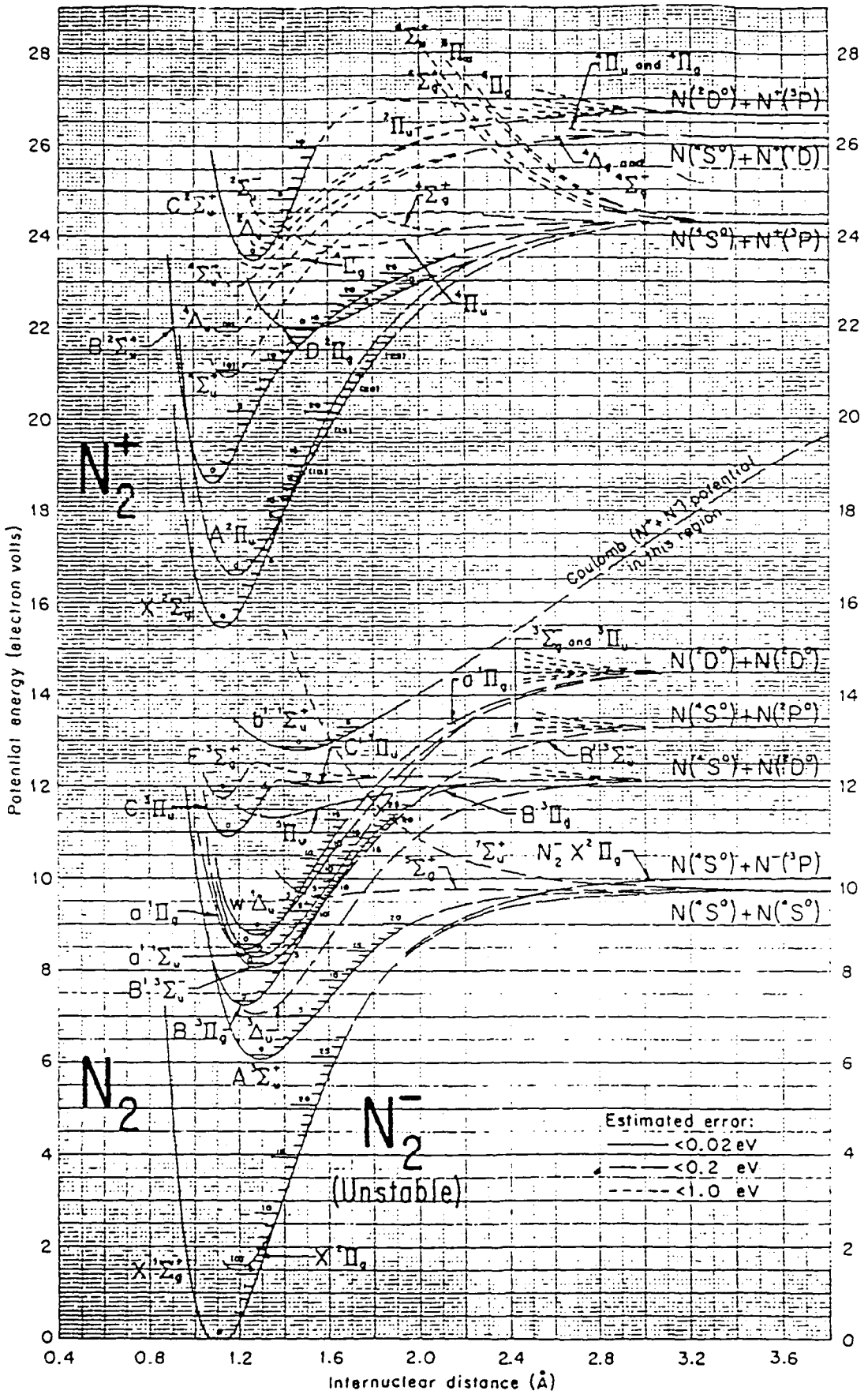


**Fig. 7 Potential-energy curves for $N_2^-$(unstable), $N_2$ and $N_2^+$**